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4 CHEMICALS AND REAGENTS

- **A.** All chemicals and commercial reagents are labeled when received with the date received and the initials of the individual receiving them, and again when opened. All chemicals and commercial reagents are placed onto an inventory list maintained either within the section or within the laboratory. All chemicals and commercial reagents are replaced when their stated shelf life has expired <u>or</u> when they fail to perform adequately under controlled conditions.
- **B.** The purity of extraction solvents, such as pentane or carbon disulfide, is critical. As a check, an appropriate amount of solvent from each lot is concentrated by evaporation and analyzed by gas chromatography. The resulting data is maintained in an appropriate location within the section.
- C. All laboratory prepared reagents will be clearly labeled to reflect the reagent name, date prepared and preparer's initials. Any working bottles of reagent aliquoted from a stock bottle will be labeled in a similar manner. The log sheets in this section will specify the information that must be captured for each reagent. This will include the quality check to ensure that the reagent is functioning properly. A stock bottle of reagent may continue to be used until the aliquoted reagent fails the quality check. If this occurs, it is the examiner's responsibility to immediately dispose of the remaining reagent and to communicate this to all examiners with working bottles of the reagent. After the initial preparation, reagents will be checked at least once per each case analyzed. The results of the QC check are recorded in the case notes. (A notation such as "QC √+" is sufficient to document the reagent reacted as expected.)
- **D.** All other reagents used in non-routine examinations, such as general chemical type cases, are prepared as necessary and are tested with knowns and blanks, the results of which are recorded in the case file notes.
- **E.** Test strips are checked with appropriate standards and results are recorded in case notes. Test strips may continue to be used past their stated expiration date as long as the QC check is positive.

F. Reagent Recipes for Routine Examinations

Aqueous Aniline Sulfate: Dissolve 5.0 g aniline sulfate in 100 ml deionized water. (Ref. 2)

Barium Chloride: Dissolve 5.0 g barium chloride in 100 ml deionized water (5% solution). (Ref.1)

Brucine: Dissolve 5.0 g brucine sulfate in 100 ml concentrated sulfuric acid. (Ref. 1)

Diphenylamine: Dissolve 0.68 g diphenylamine in 45 ml concentrated sulfuric acid. Place in ice bath and

cautiously add 22.5 ml glacial acetic acid. (Ref. 6)

Hydrochloric Acid (15%): Mix 7.9 ml concentrated HCl into 12.1 ml deionized water. (Ref. 7)

LeRosen: Mix 75 ml of concentrated sulfuric acid in 1.5 ml of 37% formaldehyde. (Ref. 3, pp.137)

1-Naphthol: Dissolve 15 g 1-naphthol in 100 ml of ethanol (Ref. 2)

Nessler: Dissolve 20 g potassium hydroxide in 50 ml deionized water. Dissolve 10 g mercuric iodide

and 5 g potassium iodide in 50 ml deionized water. Mix together. (Ref. 4)

Silver Nitrate: Dissolve 3.75 g silver nitrate in 75 ml deionized water. (Ref. 1)

Sodium Hydroxide: 2.0 N: Dissolve 6.0 g NaOH in 75 ml deionized water. (Ref. 4, pp. 57, 64)

Saturated: Dissolve 10g NaOH in 100 ml deionized water (Ref. 2)

Sulfuric Acid (75%): Mix 56.25 ml concentrated sulfuric acid into 18.75 ml deionized water. (Ref. 7)

Triphenyl Selenium Chloride: Saturated solution in 60 ml deionized water. (Ref. 5)

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G. Reagent Recipes for Non-Routine Examinations

Fehling's: Dissolve 7.5 g copper sulfate in 100 ml deionized water. Dissolve 35 g sodium tartrate and 25

g potassium hydroxide in 100 ml deionized water. Mix equal volumes of each solution. (Ref.

3, pp.131)

Nitron: Dissolve 3.75 g nitron (diphenylenedianilohydrotriazole) in 75 ml of 88% formic acid.(Ref. 1)

Triphenyltetrazolium Chloride: Dissolve 0.38 g triphenyltetrazolium chloride in 75 ml deionized water. (Ref. 3, pp. 338)

H. References

- 1. Parker, R.G., Stephenson, M.O., McOwen, J.M, Cherolis, J.A., "Analysis of Explosives and Explosive Residues. Part 1: Chemical Tests", Journal of Forensic Sciences, 1975, 20, 1, pp. 133-140.
- 2. "Spot Tests, Systematic Analysis Of Low Explosives", Bureau of Alcohol, Tobacco, and Firearms, Rev 6/88.
- 3. Feigl, F. "Spot Tests In Organic Analysis" 7th Ed, 1966.
- 4. Jungreis, Ervin.; "Spot Test Analysis, Clinical, Environmental, Forensic, and Geochemical Applications", Volume 75, 1985, pp.57-58.
- 5. Anger, V., and Feigl, F., Spot Tests in Inorganic Analysis, 6th ed., Elsevier Publishing Company: Amsterdam, The Netherlands, 1972, p.184.
- 6. Thornton, John I., "Forensic Paint Examination," Forensic Science Handbook, Volume 1, Prentice-Hall, Inc., 1982, p.550.
- 7. Hall, David.; "Practical Fiber Identification"; Textile Engineering Department, Auburn University., 1976; p.36.

I. Reagent Log Sheets (see following pages)

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REAGENTS LOG SHEET

AQUEOUS ANILINE SULFATE

Dissolve 5.0 g aniline sulfate in 100 ml deionized water.

A drop of aniline sulfate is added to the test solution. Two drops of sulfuric acid are added. A blue ring at the aqueous/acid interface indicates the presence of chlorate ions.

Check against potassium chlorate. Positive is a blue color.

Date	Initials	Aniline Sulfate – Mfg/Grade	Lot#	QC √

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REAGENTS LOG SHEET

BARIUM CHLORIDE

Dissolve 5.0 g barium chloride in 100 ml deionized water.

Check against potassium sulfate. Positive is a white precipitate.

Date	Initials	Barium Chloride - Mfg/Grade	Lot#	QC √

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REAGENTS LOG SHEET

BRUCINE

Dissolve 5.0 g brucine sulfate in 100 ml concentrated sulfuric acid.

Check against potassium nitrate. Positive is an orange to yellow color.

Date	Initials	Brucine Sulfate – Mfg/Grade	Lot#	QC √

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REAGENTS LOG SHEET

DIPHENYLAMINE

Dissolve 0.68 g diphenylamine in 45 ml concentrated sulfuric acid. Place in ice bath and cautiously add 22.5 ml glacial acetic acid.

Check against potassium nitrate. Positive is a blue to blue-black color.

Date	Initials	DPA - Mfg/Grade	Lot#	Conc.H ₂ SO ₄ - Mfg/Grade	Lot#	Glacial Acetic Acid - Mfg/Grade	Lot#	QC √

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REAGENTS LOG SHEET

FEHLING'S

Dissolve 7.5 g copper sulfate in 100 ml deionized water. Dissolve 35 g sodium tartrate and 25 g potassium hydroxide in 100 ml deionized water. Mix equal volumes of each solution.

Check against known lactose and sucrose. Positive is a yellow/orange/red precipitate.

Date	Initials	Copper Sulfate - Mfg/Grade	Lot#	Na Tartrate - Mfg/Grade	Lot#	KOH - Mfg/Grade	Lot#	QC √

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REAGENTS LOG SHEET

15% HYDROCHLORIC ACID

Mix 7.9 ml concentrated HCl into 12.1 ml deionized water.

Check against known nylon 6 and nylon 66 fibers. Positive is nylon 6 fiber dissolves, nylon 66 fiber is insoluble.

Date	Initials	Conc. HCl - Mfg/Grade	Lot#	QC √

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REAGENTS LOG SHEET

LeROSEN

Mix 75 ml of concentrated sulfuric acid in 1.5 ml of 37% formaldehyde.

Check against toluene. Positive is a red color.

Date	Initials	Conc. Sulfuric Acid - Mfg/Grade	Lot#	37% Formaldehyde - Mfg/Grade	Lot#	QC √

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REAGENTS LOG SHEET

1-NAPHTHOL

Mix 15 g of 1-naphthol in 100 mls of ethanol.

One or two drops of the unknown solution is placed in a white spot plate. Add one drop of naphthol solution and two drops of concentrated sulfuric acid. The appearance of a blue or purple-blue color indicates the presence of sugar (sucrose). Check with sucrose.

Notes: Fructose forms a similar purple-blue color. Glucose and maltose form pink colors at the sulfuric acid interface. Keep reagent from light. Nitrates form a yellow-green color which obscures the color test.

Date	Initials	1-Naphthol - Mfg/Grade	Lot#	Ethanol - Mfg/Grade	Lot#	QC √

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REAGENTS LOG SHEET

NESSLER

Dissolve 20 g potassium hydroxide in 50 ml deionized water. Dissolve 10 g mercuric iodide and 5 g potassium iodide in 50 ml deionized water. Mix together.

Check against ammonium nitrate. Positive is an orange to brown precipitate.

Date	Initials	KOH - Mfg/Grade	Lot#	HgI ₂ - Mfg/Grade	Lot#	KI - Mfg/Grade	Lot#	QC √

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REAGENTS LOG SHEET

NITRON

Dissolve 3.75 g nitron (diphenylenedianilohydrotriazole) in 75 ml of 88% formic acid.

Check against potassium nitrate. Positive is the formation of a white precipitate, often in the form of needles.

Date	Initials	Nitron - Mfg/Grade	Lot#	88% Formic Acid - Mfg/Grade	Lot#	QC √

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REAGENTS LOG SHEET

SILVER NITRATE

Dissolve 3.75 g silver nitrate in 75 ml deionized water.

Check against sodium chloride. Positive is the formation of a white precipitate.

Date	Initials	AgNO ₃ - Mfg/Grade	Lot#	QC √

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REAGENTS LOG SHEET

75% SULFURIC ACID

Mix 56.25 ml concentrated sulfuric acid into 18.75 ml deionized water.

Check against a known nylon fiber. Positive is fiber dissolves.

Date	Initials	Initials Conc. H ₂ SO ₄ - Mfg/Grade		QC √

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REAGENTS LOG SHEET

TRIPHENYLSELENIUM CHLORIDE

Saturated Solution in 60 ml deionized water.

Check against potassium perchlorate. Positive is the formation of a white precipitate.

Date	Initials	TPS Cl - Mfg/Grade	Lot#	QC √

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REAGENTS LOG SHEET

TRIPHENYLTETRAZOLIUM CHLORIDE (0.5 N SODIUM HYDROXIDE)

Dissolve 0.38 g triphenyltetrazolium chloride in 75 ml deionized water.

0.5 N NaOH: Dissolve 1.5 g NaOH in 75 ml deionized water.

Check against lactose and sucrose. Positive is a red color or red precipitate.

Date	Initials	Triphenyltetrazolium Cl - Mfg/Grade	Lot#	NaOH - Mfg/Grade	Lot#	QC \